

**Chemsearch Journal 1(1): 8 - 9**

Received: October, 2009

Accepted: May, 2010

Publication of Chemical Society of Nigeria, Kano Chapter

A STUDY OF THE KINETIC OF SYNTHESIS AND CROSSLINKING OF METHYLOL MELAMINE

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One mole of melamine (2,4,6 – amino 1,3,5 – triazine), approximately 126 g were reacted separately in suitable beakers on hot plate with stirrer using 144 cm³, 216 cm³, 288 cm³, 360 cm³ and 432 cm³, of 37% formaldehyde which is equivalent to 2, 3, 4, 5 and 6 moles respectively. The reaction was carried out in the presence of little quantity sodium dihydrogen phosphate as a catalyst, at various temperatures, molar ration of the reactants and concentration of catalyst. Since melamine is hexafunctional, complete formylation produces up to hexamethylol derivative before crosslinking take place. The kinetic of the reaction were affected by varying these parameters but follows a none complicated form. The relative viscosity of each resin increase as concentration increases. Increase in the amount of catalyst used and temperature of reaction medium reduced the time of formation and subsequent crosslinking to the resins.

Keywords: Kinetic, Synthesis, Melamine

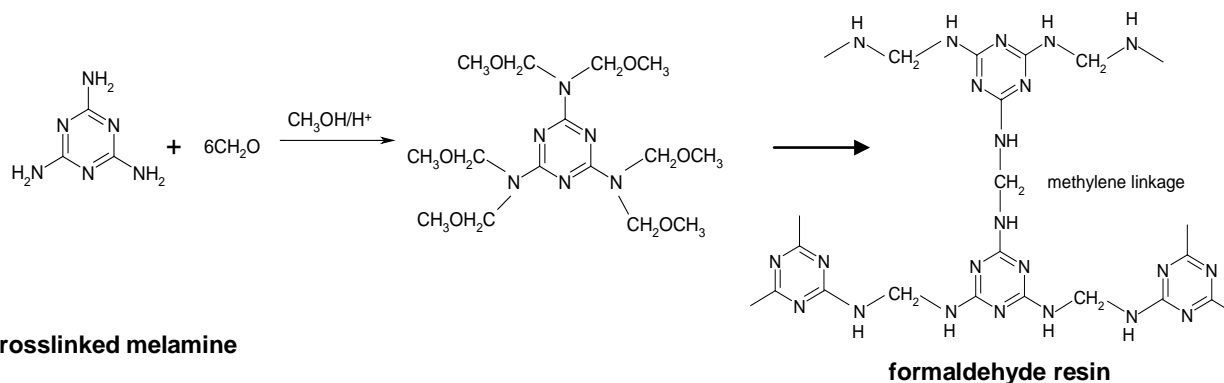
INTRODUCTION

Melamine formaldehyde resins are much more of industrial importance because of its resistance to heat, water chemical effects (Young and Lovell, 1992). These resins, which are in the class of amino resins, have wide range of applications compared to phenolic resins. These include; textile finishing, paper treatment, laminates, leather tanning to mention but a few. Formylation of melamine to obtain these products follows a complex pattern. The melamine reacts with formaldehyde, first by nucleophilic addition to form methylol compounds. In most cases di-,tri tetra-, penta-, hexa-methylol derivatives are obtained (Fried, 2000). Since melamine is not readily soluble in water or formaldehyde at room temperature, it is sometimes, necessary to heat the reaction mixture to about 80°C to get the methylol compounds before crosslinking to form the resins (Sharma 2006). Isolating the methylol compounds is a complex chemistry as the prepolymers immediately crosslinked by polycondensation to form network of rigid polymer which are insoluble in many solvents. While the chemistry of the formylation of melamine and subsequent crosslinkage of the prepolymer into a rigid network are well document (Ajayi 2004, Trotman 1975), the knowledge of the kinetics and thermodynamics involved remain inadequate in literature. Also kinetic data on synthesis and crosslinking of methylol melamine are scarce.

Although Igwe and Ogbobe (2001) have carried out a study on the kinetics of alkyd resins formation using glycerol and phthalic anhydride in the presence of lead oxide and using water evolved during the reaction and fall in acid value to establish the kinetics as second order generally at degree of polymerization greater than 5. The objective of this study is therefore to examine the influence some of the physiochemical parameters on the formation of melamine formaldehyde resins using kinetic expressions.

EXPERIMENTAL

Simple approaches to the kinetic study were carried out in 100cm³ capacity beaker placed on hot plate with magnetic stirrer i.e. in a similar way to the procedure described by Ajayi *et al* (2001) for methylol urea. The melamine and formaldehyde were mixed in molar ratio 1:2 respectively with little quantities of sodium dihydrogen phosphate (0.01g). The reaction temperature was initially at temperature of 30°C then 40°C, 60°C and 80°C respectively. The time taken to obtain a white viscous check liquid is taken as the point at which the prepolymer must have crosslinked. The experiment was repeated using 3, 4, 5 and 6 moles formaldehyde respectively and with varying and with varying amount of sodium dihydrogen phosphate; 0.05g, 0.025g, and 0.01g accordingly. The experiment is generally represented below by the equation below

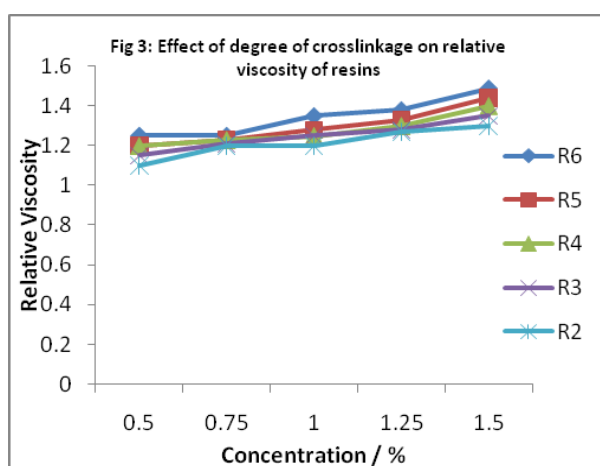
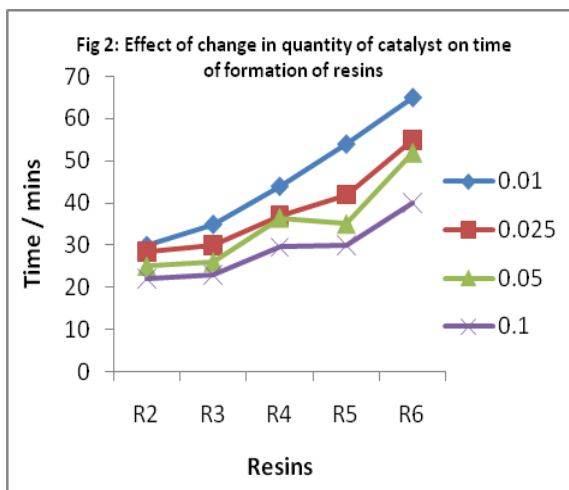
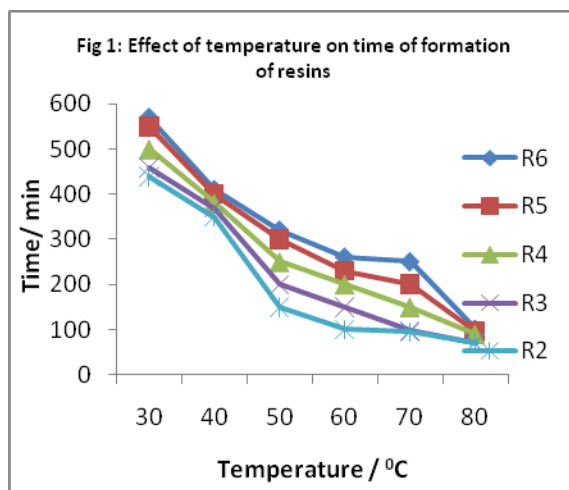


RESULT AND DISCUSSION

$$\frac{d[\text{resins}]}{dt} = k \quad d[\text{resins}] = kdt \quad \int d[\text{resins}] = \int kdt \quad [\text{resins}] = kt$$

Since the melamine is neither soluble in water nor the formaldehyde, keeping the reaction mixture at 80°C increase the rate of the crosslinking in the formation of each resins. Also, varying the amount of catalyst used at this temperature increase the rate of formation of each of the resins as shown in Fig. 2. The relative viscosity of each resins increases as concentration

increases and also as the number of methylol group in the prepolymers increases. For the fact that the viscosity of each resins increases as the methylol groups in the prepolymers increase as shown in Fig. 3, it is expected that the molecular mass will increase from R2 to R6 (Young and Lovell, 1992).



Figures 1 - 3 : shows that rate of formation increase as the temperature of reaction increases.

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